

इंटरनेट

मानक

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IS 8496 (1977): Propoxur, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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Indian Standard
SPECIFICATION FOR
PROPOXUR, TECHNICAL

(First Reprint OCTOBER 1989)

UDC 632.951 PRO

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAMAN MARG,
NEW DELHI 110002

(Continued from page 1)

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(Continued on page 7)

AMENDMENT NO. 1 NOVEMBER 1978
TO
IS : 8496-1977 SPECIFICATION FOR
PROPOXUR, TECHNICAL

Alteration

(Pages 5 and 6, Appendix A) — Substitute the following for the existing matter:

APPENDIX A
[Table 1, Item (i)]

DETERMINATION OF PROPOXUR CONTENT

A-0. GENERAL

A-0.1 For the determination of propoxur content, two methods have been prescribed. Either of these can be used as routine method.

A-1. HYDROLYSIS METHOD

A-1.1 Principle — The sample is hydrolysed with 60 percent sulphuric acid under reflux and the methylamine formed is distilled into 2 percent boric acid solution and titrated directly against standard 0.1 N sulphuric acid using bromocresol green indicator.

A-1.2 Reagents

A-1.2.1 Sulphuric Acid Solution — 60 percent (*m/v*). Prepare by adding 33 ml of concentrated sulphuric acid to 50 ml of distilled water. Mix and allow to cool. Make up to 100 ml with distilled water.

A-1.2.2 Boric Acid Solution — 2 percent (*m/v*). Prepare by dissolving 20 g boric acid in about 500 ml of hot water, cool, add 2 ml of 0.1 percent bromocresol green, dilute to one litre, and mix. This solution need not be prepared accurately and may be prepared in a large volume sufficient for several weeks' use. If the colour fades on standing, more indicator may be added to the remaining portion.

A-1.2.3 Sodium Hydroxide Solution Concentrated — Add 40 g of sodium hydroxide to about 75 ml of distilled water. Mix and allow to cool. Make up to 100 ml with distilled water.

A-1.2.4 Bromocresol Green Indicator Solution — 1 percent (*m/v*). Dissolve bromocresol green in ethanol, alternatively prepare an aqueous solution of the sodium salt as follows.

A-1.2.4.1 Rub 100 mg of bromocresol green in an agate mortar with 2.9 ml of 0.05 N sodium hydroxide. After the indicator has dissolved completely, add sufficient water to make the volume up to 100 ml.

A-1.3 Procedure — Weigh accurately about 0.8 g of the sample into a 250-ml round bottomed flask fitted with a ground-glass stopper, add 50 ml of sulphuric acid solution, heat under reflux for one and a half hours, then add 10 ml of sulphuric acid solution and reflux for another half an hour. Cool and transfer the digested sample quantitatively to a one-litre flask. Add 600 ml of the distilled water and 100 ml of concentrated sodium hydroxide solution. Immediately connect the flask to a condenser (all glass) and distil the liberated methylamine into 750-ml flask containing 50 ml of boric acid solution and bromocresol indicator, until 300 to 350 ml of liquid are collected. Remove the receiver and titrate the contents with 0.1 N standard sulphuric acid until the solution has been brought back to its original yellow-green colour.

A-1.4 Calculation

$$\text{Propoxur content, percent by mass} = \frac{V N \times 20.9}{M}$$

where

V = volume, in ml, of 0.1 N standard sulphuric acid required to titrate the steam-distilled methylamine;

N = normality of standard sulphuric acid; and

M = mass, in g, of sample taken for the test.

A-2. SAPONIFICATION METHOD

A-2.1 Principle — Propoxur is saponified with potassium hydroxide in diethylene glycol and the methyl amine formed is directly absorbed in boric acid solution and titrated against standard hydrochloric acid.

A-2.2 Apparatus — Assemble the apparatus as shown in Fig. 1.

A-2.3 Reagents

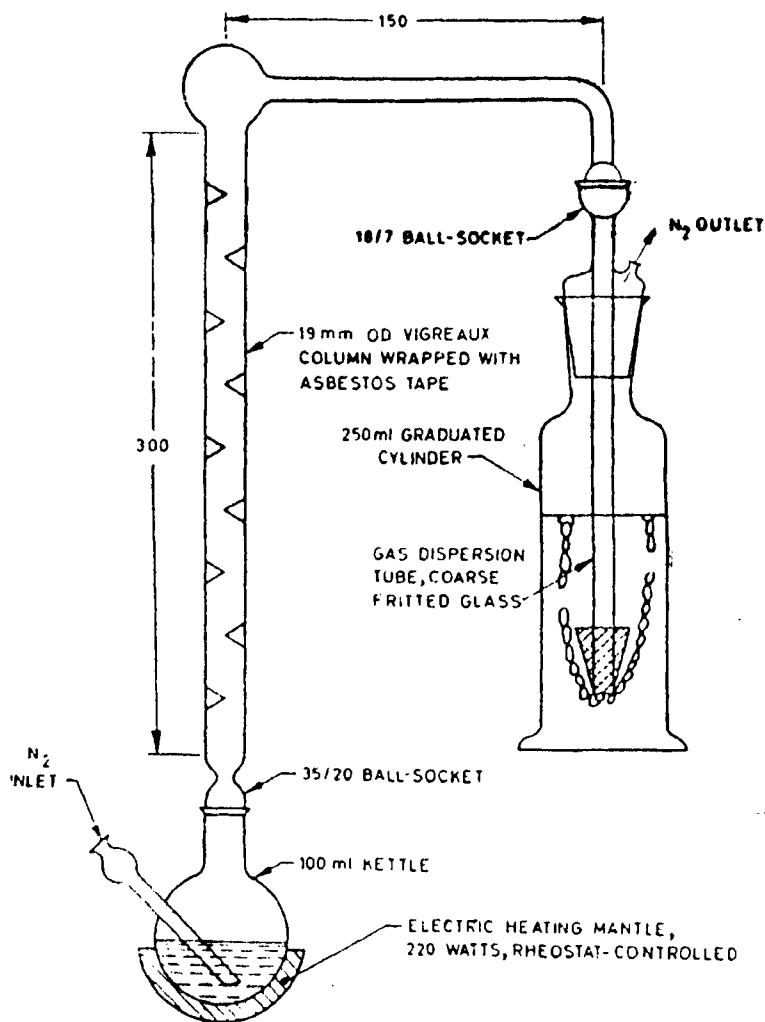
A-2.3.1 Potassium Hydroxide in Diethylene Glycol — 2 N. Dissolve 56 g of potassium hydroxide in a minimum quantity of water and make up to 500 ml with diethylene glycol.

A-2.3.2 Boric Acid Solution — See A-1.2.2.

A-2.3.3 Nitrogen

A-2.3.4 Bromocresol Green Indicator Solution — See A-1.2.4.

A-2.3.5 Hydrochloric Acid — 0.1 N.



All dimensions in millimetres.

FIG. 1 APPARATUS FOR DETERMINATION OF PROPOXUR CONTENT

A-2.4 Procedure — Weigh accurately about 0.5 g of propoxur in the 100-ml. reaction flask. Add 50 ml of potassium hydroxide in diethylene glycol and connect the flask to the evolution assembly and nitrogen. Place 100 ml of boric acid solution in the 250-ml measuring cylinder and connect the same to the gas dispersion tube. Connect nitrogen from

cylinder and adjust the flow rate to 150 ml/minute. Start heating the flask and attain boiling within 5 minutes. Continue heating and passing nitrogen for a period of one and a half hours. Disconnect the cylinder containing boric solution and transfer into a 250-ml conical flask. Wash the cylinder and the dispersion tube with distilled water and add the washings to the contents of the conical flask. Titrate immediately against 0.1 N hydrochloric acid. The end point is from green to yellow.

A-2.5 Calculation

$$\text{Propoxur content, percent by mass} = \frac{V \times N \times 20.9}{M}$$

where

V = volume of standard hydrochloric acid consumed in the test;

N = normality of hydrochloric acid; and

M = mass of sample, in g, taken for the test.

(AFCD 6)

AMENDMENT NO. 2 OCTOBER 1979
TO
IS:8496-1977 SPECIFICATION FOR PROPOXUR, TECHNICAL
Alteration

[Page 4, Table 1, col 3, against Sl No.(1)] -
Substitute '95' for '98'.

(AFCD 6)

Reprography Unit, BIS, New Delhi, India



AMENDMENT NO. 3 MAY 1982
TO
IS : 8496-1977 SPECIFICATION FOR
PROPOXUR, TECHNICAL

Alteration

[*Pages 5 and 6, Appendix A (see also Amendment No. 1)*]— Substi-
tute the following for the existing Appendix:

APPENDIX A

[*Table 1, Item (i)*]

DETERMINATION OF PROPOXUR CONTENT

A-O. GENERAL

A-0.1 Either of the two methods, namely ultra-violet (uv) spectrophoto-
metric method (see **A-1**) or saponification method (see **A-2**) may be
used for the determination of propoxur content. However, ultra-violet
spectrophotometric method (see **A-1**) shall be the referee method in case
of dispute.

A-1. ULTRA VIOLET SPECTROPHOTOMETRIC METHOD

A-1.1 Principle — 2-Isopropoxy phenol liberated by alkaline hydrolysis
is determined from the differential ultra-violet spectrum of its alkaline
solution against acidic solution at 292 nm.

A-1.2 Apparatus

A-1.2.1 UV Spectrophotometer

A-1.2.2 Volumetric Flasks — 100 ml capacity.

A-1.2.3 Pipette — 10 ml capacity.

A-1.2.4 Cuvettes — 1 cm quartz.

A-1.3 Reagents

A-1.3.1 Ethyl Alcohol

A-1.3.2 Sodium Hydroxide Solution — 1 N.

Price Rs 1.50

A-1.3.3 Sulphuric Acid Solution — 1 N.

A-1.3.4 Propoxur — of known 99 percent plus purity.

A-1.4 Preparation of Standard and Sample Solutions — It is very important in preparing the *Standard* and *Sample Solutions* that volume fluctuations with changing temperatures are avoided. This is accomplished by bringing each solution to the volume mark just before proceeding to the next step in the procedure as indicated.

A-1.4.1 Preparation of Standard Solution

A-1.4.1.1 Accurately weigh 0.37 to 0.38 g of pure propoxur (see A-1.3.4) in a 100 ml volumetric flask. Add ethyl alcohol to dissolve and make up the volume with alcohol. Call this as *Standard Solution A*.

A-1.4.1.2 Transfer 10 ml of *Standard Solution A* into another 100 ml volumetric flask. Add 50 ml of ethyl alcohol and 2.5 ml of 1 N sodium hydroxide solution. Keep the mixture at room temperature for 1 hour. Dilute to mark with alcohol and shake well to get uniform solution. Call this as *Standard Solution B*.

A-1.4.2 Preparation of Sample Solutions

A-1.4.2.1 Accurately weigh a quantity of the sample containing 0.350 ± 0.020 g of propoxur in 100 ml volumetric flask. Add ethyl alcohol to dissolve and make up the volume. Call this as *Sample Solution A*.

A-1.4.2.2 Transfer 10 ml of *Sample Solution A* into another 100 ml volumetric flask. Add 50 ml of ethyl alcohol and 2.5 ml of 1 N sodium hydroxide solution. Keep the mixture at room temperature for one hour. Dilute to mark with ethyl alcohol and shake well to get uniform solution. Call this as *Sample Solution B*.

A-1.5 Procedure

A-1.5.0 To obtain accurate results it is important that analysis of *Standard* and *Sample Solutions* be carried simultaneously, through the procedure given in **A-1.5.1**.

A-1.5.1 Pipette out 10 ml each of *Standard Solution B* (see A-1.4.1.2) and *Sample Solution B* (see A-1.4.2.2) into two separate sets of 100 ml volumetric flasks. To one set of flasks comprising *Standard* and *Sample Solutions* add 20 ml of 1 N sulphuric acid solution while to another set of flasks add 20 ml of 1 N sodium hydroxide solution. Make up the volume in each flask with ethyl alcohol. After thorough mixing take the UV transmittance/absorbance curve from 275 to 325 m μ , taking acidic solution in reference cell and alkaline solution in sample cell. The maximum absorption will be at around 292 nm.

NOTE — The absorbance measurement of the *Standard* and *Sample Solutions* shall agree within ± 10 percent of each other.

A-1.6 Calculation

A-1.6.1 Propoxur content, percent
by mass
$$= \frac{A_2}{A_1} \times \frac{M_1}{M_2} \times P$$

where

A_1 = absorbance of standard solution;

A_2 = absorbance of sample solution;

M_1 = mass, in g, of pure propoxur;

M_2 = mass, in g, of sample taken for test; and

P = percent purity of pure propoxur.

A-2. SAPONIFICATION METHOD

A-2.1 Principle — Propoxur is saponified with potassium hydroxide in diethylene glycol and the methyl amine formed is directly absorbed in boric acid solution and titrated against standard hydrochloric acid.

A-2.2 Apparatus — Assemble the apparatus as shown in Fig. 1.

A.2.3 Reagents

A-2.3.1 Potassium Hydroxide in Diethylene Glycol — 2 N. Dissolve 56 g of potassium hydroxide in a minimum quantity of water and make up to 500 ml with diethylene glycol.

A-2.3.2 Boric Acid Solution — 2 percent (*m/v*). Prepare by dissolving 20 g boric acid in about 500 ml of hot water, cool, add 2 ml of 0.1 percent bromocresol green, dilute to one litre, and mix. This solution need not be prepared accurately and may be prepared in a large volume sufficient for several weeks' use. If the colour fades on standing, more indicator may be added to the remaining portion.

A-2.3.3 Nitrogen

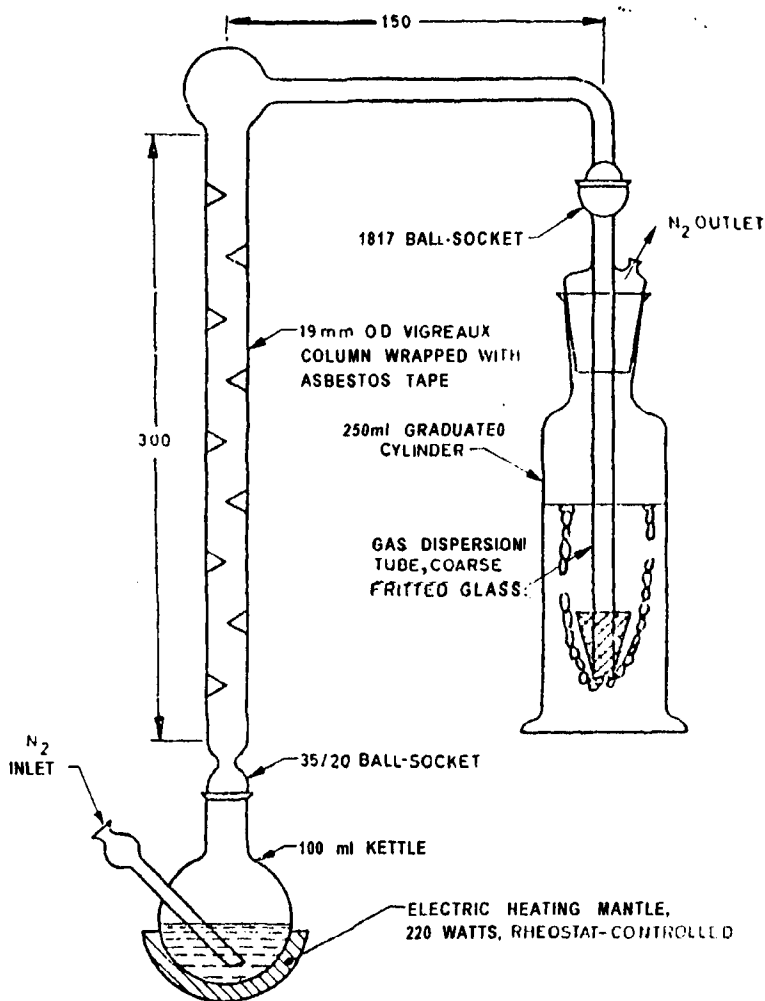
A-2.3.4 Bromocresol Green Indicator Solution — 1 percent (*m/v*). Dissolve bromocresol green in ethyl alcohol alternatively prepare an aqueous solution of the sodium salt as follows.

A-2.3.4.1 Rub 100 mg of bromocresol green in an agate mortar with 2.9 ml of 0.5 N sodium hydroxide. After the indicator has dissolved completely, add sufficient water to make the volume up to 100 ml.

A-2.3.5 Hydrochloric Acid — 0.1 N.

A-2.4 Procedure — Weigh accurately about 0.5 g of propoxur in the 100-ml reaction flask. Add 50 ml of potassium hydroxide in diethylene glycol and connect the flask to the evolution assembly and nitrogen. Place 100 ml of boric acid solution in the 250-ml measuring cylinder and connect the same to the gas dispersion tube. Connect nitrogen from

cylinder and adjust the flow rate to 150 ml/minute. Start heating the flask and attain boiling within 5 minutes. Continue heating and passing nitrogen for a period of one and a half hours. Disconnect the cylinder containing boric solution and transfer into a 250-ml conical flask. Wash the cylinder and the dispersion tube with distilled water and add the washings to the contents of the conical flask. Titrate immediately against 0.1 N hydrochloric acid. The end point is from green to yellow.



All dimensions in millimetres.

FIG. 1 APPARATUS FOR DETERMINATION OF PROPOXUR CONTENT

A-2.5 Calculation

$$\text{Propoxur content, percent by mass} = \frac{V \times N \times 20.9}{M}$$

where

V = volume of standard hydrochloric acid consumed in the test;

N = normality of hydrochloric acid; and

M = mass of sample, in g, taken for the test.

(AFCDG 6)

AMENDMENT NO. 4 DECEMBER 1988

TO

**IS : 8496-1977 SPECIFICATION FOR
PROPOXUR, TECHNICAL**

*(P&e 6, clause A-1.3.1 (see also Amendment No. 3)]— Substitute
' Methyl Alcohol ' for ' Ethyl Alcohol '.*

*[Page 6, clause A-1.3.2 (see also Amendment No. 3)]— Substitute
' Sodium Methoxide Solution —1 N. 'for ' Sodium Hydroxide Solution — 1 N. '.*

(AFCDC 6)

Printed at New India Printing Press. **Khurja**, India

AMENDMENT NO. 5 JULY 1990
TO
IS 8496 : 1977 SPECIFICATION FOR PROPOXUR,
TECHNICAL

[*Page 6, clause A-1.4 (see also Amendment No. 3)*] — Substitute the following for the existing clause:

‘A-1.4 Preparation of Standard, Sample and Blank Solutions’

[*Page 6, clause A-1.4.2.2 (see also Amendments No. 3 and 4)*] — Insert the following after A-1.4.2.2:

‘A-1.4.3 *Preparation of Blank Solution* — Prepare a blank solution exactly in the same manner as described in A-1.4.2.1 and A-1.4.2.2.’

[*Page 6, clause A-1.5.1 (see also Amendments No. 3 and 4)*] — Substitute the following for the existing clause:

‘A-1.5.1 Take out with the help of pipettes **10 ml** each of *Standard Solution B* (see A-1.4.1.2) and *Sample Solution B* (see A-1.4.2.2) into two separate sets of **100-ml** volumetric flasks. To one set of flasks comprising *Standard and Sample Solutions* add **20 ml** 1N sulphuric acid solution while to another set of flasks add 20 ml 1N sodium methoxide solution. Make up the volume in each flask with methyl alcohol. Similarly **prepare** acidic and alkaline solutions of the *Blank Solution* (see A-1.4.3). Use the acidic solution in the reference cell and the alkaline solution to adjust the zero of the spectrophotometer. After thorough mixing separately of the *Standard Solution* and *Sample Solution*, take the absorbance curve from 292 to 294 mm.

NOTE — The absorbance measurement of the *Standard and Sample Solutions* should agree within ± 10 percent each other.’

(FADC 1)

Indian Standard

SPECIFICATION FOR PROPOXUR, TECHNICAL

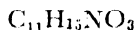
0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 June 1977, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

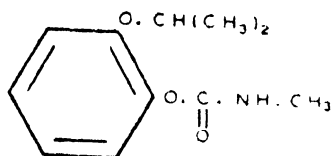
0.2 Propoxur is a non-systemic insecticide and is effective against agricultural and household insect pests.

0.3 Propoxur is the accepted common name by the International Organization for Standardization (ISO) for the insecticidal chemical *O*-isopropoxyphenyl methyl carbamate or 2-isopropoxyphenyl methyl carbamate. The empirical and structural formulae and the molecular mass of propoxur are as indicated below:

Empirical Formula



Structural Formula



Molecular Mass

209

0.4 In the preparation of this standard, due consideration has been given to the provisions of the Insecticides Act, 1968, and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS:2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for propoxur, technical.

2. REQUIREMENTS

2.1 Description — Propoxur, technical, shall be in the form of white to light pink crystalline solid, with a characteristic odour, and shall be free from foreign matter or added modifying agents.

2.2 The material shall also comply with the requirements specified in Table 1.

TABLE 1 REQUIREMENTS FOR PROPOXUR, TECHNICAL

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO	
			Appendix of this Standard	Cl No. of IS : 6940-1973*
(1)	(2)	(3)	(4)	(5)
i)	Propoxur content, percent by mass, <i>Min</i>	98	A	—
ii)	Moisture content, percent by mass, <i>Max</i>	0.5	—	4
iii)	Material insoluble in acetone, percent by mass, <i>Max</i>	0.1	—	9
iv)	Melting point	86 to 91.5°C	—	6
v)	Acidity (as H_2SO_4), percent by mass, <i>Max</i>	0.05	—	11.3

*Methods of tests for pesticides and their formulations.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in clean and dry mild steel containers with a loose polyethylene liner of thickness not less than 0.062 mm. The containers shall also comply with general requirements as stipulated in 2 of IS : 8190 (Part I)-1976*.

3.2 Marking — The container shall bear legibly and indelibly the following information and any other information as is necessary under the Insecticides Act and Rules:

- Name of the material;
- Name of the manufacturer;
- Batch number;
- Date of manufacture;
- Propoxur content, percent (*m/m*);

*Requirement for packing of pesticides: Part I Solid pesticides.

- f) Net mass of contents; and
- g) Minimum cautionary notice worded as in the Insecticides Act and Rules.

3.2.1 Each container may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 'Indian Standard methods for sampling of pesticides and their formulations' (under preparation).

NOTE — Till such time the standard under preparation is published, the matter shall be as agreed to between the concerned parties.

5. TESTS

5.1 Tests shall be carried out by the appropriate methods referred to in col 4 and 5 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977*) shall be employed in the tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[Table 1, Item (i)]

DETERMINATION OF PROPOXUR CONTENT

A-0. PRINCIPLE OF THE METHOD

A-0.1 The sample is hydrolysed with 60 percent sulphuric acid under reflux and the methylamine formed is distilled into 2 percent boric acid solution and titrated directly against standard 0.1 N sulphuric acid using bromocresol green indicator.

*Specification for water for general laboratory use (second revision).

A-1. REAGENTS

A-1.1 Sulphuric Acid Solution — 60 percent (m/v). Prepare by adding 33 ml of concentrated sulphuric acid to 50 ml of distilled water. Mix and allow to cool. Make up to 100 ml with distilled water.

A-1.2 Boric Acid Solution — 2 percent (m/v). Prepare by dissolving 20 g boric acid in about 500 ml of hot water, cool, add 2 ml of 0.1 percent bromocresol green, dilute to one litre, and mix. This solution need not be prepared accurately and may be prepared in a large volume sufficient for several weeks' use. If the colour fades on standing, more indicator may be added to the remaining portion.

A-1.3 Sodium Hydroxide Solution Concentrated — Add 40 g of sodium hydroxide to about 75 ml of distilled water. Mix and allow to cool. Make up to 100 ml with distilled water.

A-1.4 Bromocresol Green Indicator Solution — 1 percent (m/v). Dissolve bromocresol green in ethanol, alternatively prepare an aqueous solution of the sodium salt as follows:

A-1.4.1 Rub 100 mg of bromocresol green in an agate mortar with 2.9 ml of 0.05 N sodium hydroxide. After the indicator has dissolved completely, add sufficient water to make the volume up to 100 ml.

A-2. PROCEDURE

A-2.1 Weigh accurately about 0.8 g of the sample into a 250-ml round bottomed flask fitted with a ground-glass stopper, add 50 ml of sulphuric acid solution, heat under reflux for one-and-a-half hours, then add 10 ml of sulphuric acid solution and reflux for another half an hour. Cool and transfer the digested sample quantitatively to a one-litre flask. Add 600 ml of distilled water and 100 ml of concentrated sodium hydroxide solution. Immediately connect the flask to a condenser (all glass) and distil the liberated methylamine into 750-ml flask containing 50 ml of boric acid solution and bromocresol indicator, until 300 to 350 ml of liquid are collected. Remove the receiver and titrate the contents with 0.1 N standard sulphuric acid until the solution has been brought back to its original yellow-green colour.

A-3. CALCULATION

$$\text{Propoxur content, percent by mass} = \frac{V N \times 20.9}{M}$$

where

V = volume, in ml, of 0.1 N standard sulphuric acid required to titrate the steam-distilled methylamine;

N = normality of standard sulphuric acid; and

M = mass, in g, of sample taken for the test.

(Continued from page 2)

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Pesticides Subcommittee, AFCDC 6 : 1

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PLANT PROTECTION ADVISER TO THE GOVERNMENT OF INDIA	Directorate of Plant Protection, Quarantine & Storage (Ministry of Agriculture & Irrigation), Faridabad
DR V. LAKSHMINARAYANA (<i>Alternate</i>)	
SHRI Y. A. PRADHAN	Rallis India Limited, Bombay
SHRI M. L. SHAH (<i>Alternate</i>)	

(Continued on page 8)

IS : 8496 - 1977

(Continued from page 7)

<i>Members</i>	<i>Representing</i>
DR V. S. RAMANATHAN	Central Revenues Control Laboratory (Ministry of Finance), New Delhi
SHRI C. DHARMA RAO	Pesticides Testing Laboratory, Hyderabad
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SHRI A. C. SHROFF	Excel Industries Limited, Bombay
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SHRI K. SUNDARAM	Hindustan Petroleum Corporation Limited, Bombay
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SHRI N. S. VENKETARAMAN	Pesticides Testing Laboratory, Madurai
DR B. L. WATTAL	National Institute of Communicable Diseases, Delhi
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†Peenya Industrial Area 1st Stage, Bangalore Tumkur Road BANGALORE 560058	{ 38 49 55 38 49 56
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53/5, Ward No. 29, R.G. Barua Road, 5th Byelane, GUWAHATI 781003	3 31 77
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Patliputra Industrial Estate, PATNA 800013	6 23 05
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Institution of Engineers (India) Building, 1332 Shivaji Nagar, PUNE 411005	5 24 35

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